IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Examiner: E. B. Elhilo; Art Unit: 1751; Docket No.: 3000

In RE: **Application of Otto Goettel, et al**

Ser. No.: **10/501,151**

Filed: **July 12, 2004**

November 2, 2006

DECLARATION OF FACTS FILED UNDER 37 C.F.R. 1.132 TO SHOW UNEXPECTED IMPROVEMENTS IN COMPARISON TO THE CLOSEST PRIOR ART

Hon. Commissioner of Patents

and Trademarks,

Washington, D.C. 20231

Sir:

In response to the Office Action dated August 2, 2006 and in addition to the accompanying amendment, please accept the following showing of experimental facts showing unexpected and surprising improvements in light fastness and bleeding of the colors of hair samples dyed with the compositions according to the above-identified U.S. Patent Application:

WHEREAS WE, Otto **GOETTEL**, citizen of Germany; Wolfram **GEIBEL**, citizen of Germany; and Emmanuel **MORAND**, citizen of Switzerland; whose post office addresses and residencies are, respectively, Route du Roule 6, 1723 Marly, Switzerland; Mazenodweg 5, D-36088 Huenfeld, Germany; and Route de Romont 15, CH-1740 Neyruz, Switzerland; have applied for Letters Patent for new and improved

N-ARYL-4, 5-DIAMINOPYRAZOLES AND COLORANTS CONTAINING THESE COMPOUNDS

in a U.S. Patent Application, Ser. No. 10/501,151, filed July 12, 2004, of which claims 1 to 10 were rejected under 35 U.S.C. 102 (b) as anticipated by U.S. Published Patent Application 2001/0009044 A1.

WHEREAS WE have quantitatively compared the dyed hair properties of hair dyed with the new substituted-4,5-diaminopyrazole compounds of new claims 11 to 20 in the accompanying amendment with the dyed hair properties of hair dyed with corresponding substituted-4,5-diaminopyrazole compounds of the closest prior art according to WO 03/008405 and DE 200 12 156 U1, and we have found that the new substituted-4,5-diaminopyrazole dye compounds according to claims 11 to 20 are unexpectedly superior for dyeing hair than the closest prior art substituted-4,5-diaminopyrazole hair dye compounds.

WHEREAS WE have quantitatively compared the light fastness and "bleeding" fastness of colors of hair dyed with colorant compositions containing the new substituted N-phenyl-4, 5-diamino-pyrazole compounds of new claims 11 to 20 with the light fastness and "bleeding" fastness of colors of hair dyed with corresponding colorant compositions containing substituted-N-phenyl-4, 5-diaminopyrazole compounds of the closest prior art according to DE 200 12 156 U1, and we have found that the colors of the hair dyed with the colorant compositions containing the new substituted N-phenyl-4, 5-diaminopyrazole hair dye compounds according to new claims 11 to 20 have unexpectedly better light fastness and resistance to diffusion, i.e. "bleeding" fastness, than the colors produced by the corresponding colorant compositions containing the substituted N-phenyl-4,5-diaminopyrazole compounds of the closest prior art.

I. HAIR DYEING METHODS

Standard animal hair samples (bleached Buffalo hair) were dyed with examples of hair colorant compositions according to the present invention as claimed in claims 11 to 20 of the accompanying amendment and also with examples of the closest prior art hair colorant compositions. Immediately prior to application, 100 g portions of a 6 % aqueous hydrogen peroxide solution were mixed with 100 g portions of each of the hair colorant compositions to obtain ready-to-apply hair dyeing mixtures. Amounts of these hair dyeing mixtures that were sufficient to dye the hair samples were applied to them. The hair dyeing

mixtures were then allowed to act on the hair samples for an acting time of 30 minutes at 40°C. Then all the dyed hair samples were washed with a shampoo, rinsed with water and dried. The color of each of the dyed hair samples was measured quantitatively with a colorimeter made by Minolta, the Minolta Chromameter Type 300. The measured color parameters in the Lab system are tabulated in the Tables appearing below.

II. THE TESTED HAIR COLORANT COMPOSITIONS

General Formulation of the Tested Hair Colorant Compositions

10.00 g	sodium lauryl ether sulfate, 28 % aqueous solution
7.85 g	ethanol
0.30 g	ascorbic acid
0.40 g	sodium sulfite
9.10 g	ammonia, 25 % aqueous solution
2.5 mmol	pyrazole derivative An according to the following list
2.5 mmol	5-amino-2-methylphenol
to 100.00 g	water

List of Pyrazole Derivatives of the Present Invention and of the Prior Art

A1(invention)	1-(2,4-dimethylphenyl)-4,5-diamino-1H-pyrazole dihydrochloride
A2(invention)	1-(2,5-dimethylphenyl)-4,5-diamino-1H-pyrazole dihydrochloride
A3(invention)	1-(4-aminophenyl)-4,5-diamino-1H-pyrazole sulfate (1:1)
A4(prior art)	1-(4'-methylphenyl)-4,5-diamino-1H-pyrazole dihydrochloride
A5(prior art)	1-(4'-methoxyphenyl)-4,5-diamino-1H-pyrazole dihydrochloride
A6(prior art)	1-(4'-chlorophenyl)-4,5-diamino-1H-pyrazole hemisulfate

III. GENERAL COLOR PARAMETER MEASUREMENT PROTOCOL

The color parameter values L_0, a_0 and b_0 of each hair strand sample after dyeing it with one of the above-described ready-to-apply hair dyeing mixtures of the claimed invention or of the prior art were measured with the Minolta Colorimeter. Then the color parameter values L_1, a_1 and b_1 for each dyed hair sample were measured after either exposure to light or "bleeding" treatment. The color difference parameters, ΔE (a measure of the overall color change), of the dyed hair samples after washing, exposure to light or sweat and "bleeding" treatment were calculated from the measured values L_0, a_0, b_0 and L_1, a_1 and b_1 according to the following formula (I):

$$(I) \quad \Delta E = \{(L_0 - L_1)^2 + (a_0 - a_1)^2 + (b_0 - b_1)^2\}^{1/2}$$

wherein L_0, a_0 and b_0 are the L value, the a value and the b value for the freshly dyed hair samples respectively, and

wherein L_1, a_1 and b_1 are the L value, the a value and the b value for the dyed hair samples after treatment, i.e. after exposure to light or "bleeding". The results are summarized in the Tables I and II hereinbelow.

IV. INDIVIDUAL COMPARATIVE EXPERIMENTS AND TABULATED MEASURED COLOR PARAMETERS

1. Light Fastness Experiments

The dyed hair samples were mounted on a cardboard support, divided in half and subsequently illuminated in an Atlas Xenotest. To simulate the light of the sun an Atlas Suntest CPS+ light device, which is equipped with a Xenon 1.5 kW lamp that produces 520 ± 10 W/m^2 (300 to 800 nm), was used. The temperature in the test chamber was 40°C during irradiation. The irradiation of the hair lasted 48 hours, which corresponded to about six weeks of exposure to the sun. Subsequently the color parameters in the Lab color system were measured. The color change difference ΔE (root mean square difference between the values measured before and after irradiation) was calculated from the measured parameters. The results are tabulated in Table I below.

TABLE I. L, a, b and ΔE Values for Dyed Hair Samples before and after Light Exposure

Colorant	L	a	b	ΔE
A1 (invention, before irradiation)	44.54	45.10	43.28	
(invention, after irradiation)	49.29	36.65	41.25	9.90
A3 (invention, before irradiation)	39.68	46.98	38.00	
(invention, after irradiation)	43.17	38.21	36.34	9.58
A5 (not the invention, before irradiation)	43.33	35.51	21.15	
(not the invention,after irradiation)	37.54	42.04	34.87	16.26
A6 (not the invention, before irradiation)	39.59	44.06	39.24	
(not the invention, after irradiation)	45.97	34.61	38.87	11.41

2. Diffusion Behavior ("Bleeding") Measurements

The dyed hair samples were suspended in 300 ml demineralized water, so that the hair samples were completely under water. Then the water was stirred with a magnetic stirrer at room temperature (20 - 25°C) for five hours. Subsequently the hair samples were dried and the color parameters of the treated hair, L, a, and b, were measured with the Minolta colorimeter. Then the difference between the color measured before and after treatment was calculated from the L, a, and b values.

TABLE II. L, a, b and ΔE Values for Dyed Hair Samples before and after treatment to measure Diffusion Behavior (Bleeding)

Colorant	L	a	b	ΔE
A1 (invention, before treatment)	45.49	44.77	43.66	
(invention, after treatment)	46.73	40.82	42.11	4.42
A2 (invention, before treatment)	48.43	42.72	44.76	
(invention, after treatment)	49.81	38.81	42.01	4.98
A4 (not the invention, before treatment)	37.94	46.04	36.98	
(not the invention, after treatment)	38.77	38.58	35.28	7.70
A6 (not the invention before treatment)	35.78	42.90	32.79	
(not the invention, after treatment)	38.35	38.98	35.65	5.49

V. CONCLUSION

The tabulated results for L, a, b and ΔE clearly show that the dyed hair colors of hair samples dyed with the colorants according to the claimed invention in claims 11 to 20 in the accompanying amendment have **unexpectedly and surprisingly improved** color fastness to exposure to light and color fastness measured in "bleeding" experiments in comparison to the dyed hair colors obtained by dyeing with hair colorants of the closest prior art.

I HEREBY DECLARE AND AFFIRM THAT ALL STATEMENTS made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statement may jeopardize the validity of the above-named application, any patent issuing thereon or any patent to which this Declaration is directed.

23 November 2006

DATE

Otto **GOETTEL**

Wolfram **GEIBEL**

Emmanuel **MORAND**